Experimental Procedure To Study The Thermomechanical Properties Of New Zirconate Thermal Barrier Coating.

It comprises the various sophisticated equipments used for experimentations for the determination of the thermo mechanical properties such as hardness of bond coated specimens before and after TSRT and also hardness of Zirconate Thermal Barrier coated Specimens after TSRT, tensile strength, adhesive strength and dry abrasive wear test on bond coated specimens and other surface structural properties such as phase analysis by XRD and microstructure by SEM or OM and chemical analysis by EDAX analysis of Zirconate TBC Coated Substrate. The above said properties testing equipment details explained in the subsequent section of this chapter.

5. Specimen preparation for Plasma Spray TBC coating:

The choice of substrates and dimensions were detected by the requirements of the specific characterization technique and their suitability for the application envisaged. One flat side of the substrate was initially cleaned with acetone, subjected to grit blasting (with alumina) and degreased in ethyl alcohol. Different types of jigs and fixtures were made (in advance) to mount the specimens according to the spraying requirements. These substrates were secured tightly in suitable holders immediately after cleaning. The initial thickness of the substrate was measured on a few spots in order to measure the bond coat and the ceramic coat thickness. The substrate on the mount was finally degreased again with ethyl alcohol. These steps were followed very rigorously because any trace of contamination on the substrate was likely to provide a weak point of adhesion for the subsequent coating.

5.1. Plasma sprays coating: A bond coat of 75 to 100 µm thick commercial nickel based alloy Ni33CrAlY, AMDRY 962 (hereafter referred to as NiCrAlY) powder was spray coated onto the freshly prepared substrate using a plasma spray technique (80Kw), Switzerland make, and SULZER METCO-2000 spray systems. The desired number of passes of the plasma gun over the substrate (calculated on the basis of the desired thickness, typically the system was set to deliver a 50µm thick coating per pass) was preset in the computer controlled plasma spray system. The thicknesses of the
substrate plus the bond coat were measured at the same spots where substrate thicknesses had been measured earlier and the average bond coat thickness was ascertained. The parameters used for plasma spraying Ni33Cr10Al1 bond coat is shown in Table-5.1

The substrate was kept air cooled during spraying, the oven-dried plasma sprayable Zirconate TBC powders of the desired composition were then plasma sprayed on the bond-coated substrates. Here again, the number of passes required for deposition of a particular thickness of coating was ascertained by measuring the thickness of the ceramic coating after a single pass under the preset chosen conditions of spraying. The thickness of the coated substrate was measured(bond and ceramic coats) on the substrate The parameters used for plasma spraying of metallic bond coat and Zirconate topcoat of different TBC powders are shown in tables-5.1((La2Zr2O7 = 57%La2O3 + 43% ZrO2, Pr2Zr2O7 = 24% Pr2O3 + 76% ZrO2 )).

5.1.1. Optimization of coatings thickness: From the literature review and discussions with experts, the bond coat thickness is set to 100 ± 25µm and top coat is 225 ± 25µm as shown in table-5, because thermal barrier coatings are purely a physical deposition of the ceramic powder where in layers get built up due to solidification of the melt. Thus, it is recommended (from literature survey and opinion from experts)[183] that best results of thermal barrier coatings are achieved when bond coat is 75-125±25µm and zirconate based ceramic top coat is 250-350µm, beyond this, the adhesive strength of coating decease gradually / falls rapidly due to built up of residual stress along with thermal grown oxide with between intermetallic bond (bottom) and ceramic top coating. Therefore in the present investigation the intermetallic bond coat was 100 ± 25µm and ceramic top coat was 250 to 350µm was chosen.

Figure- 5. Shows the typical photographs of a set of mounted substrates along with a part of the robot controlled plasma spray coating, Plasma Spray is perhaps the most flexible of all of the thermal spray processes as it can develop sufficient energy to melt any material. Since it uses powder as the coating feedstock, the number of coating materials that can be used in the plasma spray process is almost unlimited. The plasma gun incorporates a cathode (electrode) and an anode (nozzle) separated by a small gap forming a chamber between the two. DC. Power (40kw) is applied to the cathode and
arcs across to the anode. And at the same time, gases are passed through the chamber. The generated powerful arc is sufficient to strip the gases of their electrons and the state of matter known as plasma is formed.

As the unstable plasma (Plasma spray is used to form deposition is greater than 50- micron of a wide range of industrial materials, including nickel and ferrous alloys, refractory ceramics, such as aluminum oxide and zirconia-based ceramics) recombines back to the gaseous state, thermal energy is released. Because of the inherent instability of plasma, the ions in the plasma plume rapidly recombine to the gaseous state and cool. At the point of recombination, temperatures can be 6,600°C to 16,600°C (12,000 °F to 30,000 °F), which exceeds the surface temperatures of the sun and it is capable of melting anything. When the coating material is injected into the gas plume, it is melted and propelled towards the target substrate. The next layer deposits onto the first immediately after and thus the coating builds up layer by layer [184,186].

Table-5. Bond and top coat thickness of used ceramic coatings.

<table>
<thead>
<tr>
<th>Sl no</th>
<th>Name of coating</th>
<th>Bond coat thickness</th>
<th>Top coat thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>La$_2$Zr$_2$O$_7$</td>
<td>100 ± 25 µm</td>
<td>250 ± 25 µm</td>
</tr>
<tr>
<td>2</td>
<td>Pr$_2$Zr$_2$O$_7$</td>
<td>100 ± 25 µm</td>
<td>250 ± 25 µm</td>
</tr>
</tbody>
</table>
Table- 5.1. Parameters for plasma spray coating of Metallic Bond coat $\text{La}_2\text{Zr}_2\text{O}_7$ topcoat and $\text{Pr}_2\text{Zr}_2\text{O}_7$ topcoat

<table>
<thead>
<tr>
<th>Sl no</th>
<th>Parameters</th>
<th>Metallic Bond coat</th>
<th>$\text{La}_2\text{Zr}_2\text{O}_7$ Top Coat</th>
<th>$\text{Pr}_2\text{Zr}_2\text{O}_7$ Top Coat</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Argon flow rate</td>
<td>65 lit/min.</td>
<td>44 lit/min.</td>
<td>44 lit/min.</td>
</tr>
<tr>
<td>2</td>
<td>Hydrogen flow rate</td>
<td>14 lit/min.</td>
<td>13 lit/min.</td>
<td>13 lit/min.</td>
</tr>
<tr>
<td>3</td>
<td>Powder gas flow rate</td>
<td>2.3 lit./ min.</td>
<td>3.3 lit./ min.</td>
<td>3.3 lit./ min.</td>
</tr>
<tr>
<td>4</td>
<td>Current</td>
<td>600 Amps.</td>
<td>630 Amps.</td>
<td>628 Amps.</td>
</tr>
<tr>
<td>5</td>
<td>Voltage</td>
<td>75 Volts</td>
<td>71 Volts</td>
<td>70 Volts</td>
</tr>
<tr>
<td>6</td>
<td>Nozzle/ Electrode diameter</td>
<td>6.0 mm</td>
<td>6.0 mm</td>
<td>6.0 mm</td>
</tr>
<tr>
<td>7</td>
<td>Injector diameter</td>
<td>1.5 mm</td>
<td>1.8 mm</td>
<td>1.8 mm</td>
</tr>
<tr>
<td>8</td>
<td>Injector angle</td>
<td>$90^\circ$</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>9</td>
<td>Injector distance</td>
<td>6.0 mm</td>
<td>8.0 mm</td>
<td>8.0 mm</td>
</tr>
<tr>
<td>10</td>
<td>Powder feed rate</td>
<td>40 gms/min.</td>
<td>40 gms/min.</td>
<td>38 gms/min.</td>
</tr>
<tr>
<td>11</td>
<td>Spray distance</td>
<td>140 mm</td>
<td>120 mm</td>
<td>120 mm</td>
</tr>
</tbody>
</table>

5.2. Characterization of metallic bond coatings:

5.2.1. Hardness tests of ceramics:

Hardness is resistance of material to Plastic deformation caused by indentation. Sometimes hardness refers to resistance of material to scratching or abrasion. In some cases relatively quick and simple hardness test may substitute tensile test. Hardness may be measured from a small sample of material without destroying it. Principle of any hardness test method is forcing an indenter into the sample surface followed by measuring dimensions of the indentation (depth or actual surface area of the indentation). Hardness of ceramics is determined by their chemical compositions and microstructure characteristics, such as porosity, grain size, grain-boundary phases. Depending on the loading force value and the indentation dimensions, hardness is defined as a macro, micro- or nano-hardness.

5.2.2. Macro-hardness tests: Macro-hardness tests are the most widely used methods for rapid routine hardness measurements. The indenting forces in macro-hardness tests are in the range of 50N to 30000N. Hardness test has been selected to find out the hardness of metallic bond coat and ceramic top coat, using micro Vickers hardness testing machine. The Vickers hardness test method consists of indenting the
test material with a diamond indenter, in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a load of 1 to 100kgf. The full load is normally applied for 10 to 15 seconds. The two diagonals of the indentation left in the surface of the material after removal of the load are measured using a microscope and their average calculated. The area of the sloping surface of the indentation is calculated. The Vickers hardness is the quotient obtained by dividing the load by the square area of indentation. The advantages of the Vickers hardness test are that extremely accurate readings can be taken, and just one type of indenter is used for all types of metals and surface treatments. Although thoroughly adaptable and very precise for testing the softest and hardest of materials, under varying loads.

The Vickers hardness test uses a square base diamond pyramid as the indenter. The included angle between the opposite faces of the pyramid is 136°. The Vickers hardness tester operates on the same basic principle as the Brinell tester, the numbers being expressed in the terms of load and area of the impression. As a result of the indenter’s shape, the impression on the surface of the specimen will be a square. The length of the diagonal of the square is measured through a microscope fitted with micrometer that contains movable knife-edges[187,188]. The Vickers hardness values are calculated by the formula:

\[
HV = \frac{2P\sin(\alpha/2)}{d^2} = 1.8544 \left(\frac{P}{d^2}\right) \text{------------------------(1)}
\]

Where \( P \) is the applied load in kg, and \( d \) is the diagonal length in mm.

### 5.2.3. Micro-Vickers Hardness Test Analysis Before Thermal Shock Test:

<table>
<thead>
<tr>
<th>Specifications Micro-Vickers Hardness tester</th>
</tr>
</thead>
<tbody>
<tr>
<td>Equipments</td>
</tr>
<tr>
<td>------------------------------------------------</td>
</tr>
<tr>
<td>Micro-Vickers Hardness tester</td>
</tr>
</tbody>
</table>

Digital micro hardness tester of load 10–1000gm load with 10 & 40x magnification and automatic turret mechanism. Fully computerized and automated software enhance the quality of the testing. This micro hardness tester is semi automatic most suitable industrial quality control and research where the limitations of small sample size persist like thin plate, foils, miniature components[189,190] etc. This above
combination of above Metallography with image analysis and digital micro hardness tester is most suitable for failure analysis of any components.

In the present study Vickers Hardness has been selected to determine hardness of 150\(\mu m\) NiCr and NiCrAl bond coated on aluminum and steel specimens, the test was conducted before and after thermal shock resistance test on bond coated specimens [191], the Vickers hardness and experimentation details are discussed in above section 5.2.1, 5.2.2, and similar test has been conducted on advanced zirconate of 100-150\(\mu m\). The detailed results of coating are discussed in subsequent chapter 8.

5.3. Adhesive Strength: A series of tensile tests are carried over the coated specimens of standard ASTM E8. Measurements were carried out using a round substrate, tensile tests are conducted on round button substrate and coated on one side and it providing controlled uniformly increasing tension force, applied to the specimen. The specimen’s one end is metallic bond coated with ceramic standard structural ceramic adhesive (resin with hardener) and other ends are gripped and fixed in the UTM machine and its gauge length \(L_0\) (a calibrated distance between two marks on the specimen surface),

And pulled under tensile load and is continuously measured by varying load until the material get rupture by the computerized UTM. A Computerized Universal Testing Machine (ASTM E8) has been used for the test, the weaker ceramic substrate interface failed and the corresponding load/area was taken as a measure of adhesive strength. Similar test has been conducted on Zirconate Thermal Barrier coated specimen and
estimate the adhesive strength, the prepared round tensile test steel and aluminum specimens to determine the adhesive strength are shown in figure 5.1 and 5.1(a). [192].

5.4. Abrasive Wear Test:

5.4.1. Wear: Wear occurs as a natural consequence when two surfaces with a relative motion interact with each other. Wear may be defined as the progressive loss of material from contacting surfaces in relative motion. The mechanism of wear is very complex and the theoretical treatment without the use of rather sweeping simplifications (as below) is not possible. When abrasion is the predominant factor causing deterioration of materials, this test will gives the data to compare materials or coatings and can help to predict the life time of a material or coating. Abrasion testing is used to test the abrasive resistance of solid materials. Materials such as metals, composites, ceramics, and thick (weld overlays and thermal spray) coatings can be tested with this method. The intent of this test method is to produce data that will reproducibly rank materials in their resistance to scratching abrasion under a specified set of conditions [193-198].

In the present experimental study, on NicrAl metallic bond coated specimen has been chosen for dry- abrasive wear test, wear properties is the predominant factor causing deterioration of coating materials, since bond coating mainly used in high temperature applications such as gas turbine, air craft, automobile diesel engines etc. to prevent wear and corrosion, also it will help good adhesion between metal substrate
and TBC, so wear properties is the main concern[199], the prepared standard rectangular shape of material with one side bond coated specimens for dry-abrasive test are shown in figure.5.2. And the detailed terminology and specifications are shown in the following table.5.3. And the experimental procedure details are shown below section 5.4.3 of this chapter.

Figure.5.2. Bond coated different specimens for dry abrasive wear test

Table: 5.3. Specifications of dry abrasive wear tester

<table>
<thead>
<tr>
<th>SI No</th>
<th>Detailed Specification</th>
<th>SI No</th>
<th>Detailed Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Material description: SS304, Al-124 uncoated, SS304, Al-124 bond coated</td>
<td>6</td>
<td>Wheel (ASTM (STD))</td>
</tr>
<tr>
<td>2</td>
<td>Specimen size: 1” width, 3” length, 0.12 to 0.5” thickness</td>
<td>7</td>
<td>Test Load: 0.5 – 12 Kgs</td>
</tr>
<tr>
<td>3</td>
<td>Leverage ratio: 1:2.5</td>
<td>8</td>
<td>Rubber wheel size (Chlorobutyl rubber lining): Ø228 mm</td>
</tr>
<tr>
<td>4</td>
<td>Speed: 200rpm</td>
<td>9</td>
<td>Abrasive sand flow rate: 200-400 gms/min</td>
</tr>
<tr>
<td>5</td>
<td>Duration: 10 Min</td>
<td>10</td>
<td>Abrasive sand (silica sand): AFS 50/70 (ASTM STD)</td>
</tr>
</tbody>
</table>

5.4.2. Evaluation of the abrasive wear resistance:

Abrasive wear resistance was evaluated using the Dry Sand/Rubber Wheel and the Dry Sand Steel/Wheel methods. Using these methods the three-body abrasive conditions are simulated. The result of this test is the materials volume loss in cubic millimeters. On the materials of the higher abrasive wear resistance, the volume loss is
lower. The test principle is schematically depicted in Figure.5. 2.1, A brief test description is as follows:

The dry rubber wheel (traditionally called Dry Sand-Rubber Wheel; DSRW) abrasion test, a modified version of ASTM G65 [200,201], was performed using alumina particles as the abrasive. This abrasive was fed between the coated or uncoated sample and the rotating rubber wheel. Specimen dimensions were 25.4mm x 76.2mm x12mm(1’’ width, 3’’ length, 0.12 to 0.5’’ thickness), the powder particle size of wear tests silica SiO$_2$ in AFS 50/70 (ASTM STD) was 100-200µm, and the samples were pressed against the rubber wheel with the force of 30N. Test duration was 10min (200 revolutions of the wheel), and All the coating weight losses were transformed to volume losses by applying densities, The materials mass loss were transformed to volume losses. The value of volume loss were/is compare with different densities of materials. The samples were weighted on digital scales with the accuracy of 0.0001g, and the similar trends of results has been found in all other selected coated and uncoated test specimens. The wear tracks in the coatings were evaluated using SEM analysis, the obtained out come results such as microstructure and sliding wear rate are discussed in chapter.8.

5.4.3. Experimental Procedure:

[i].Remove all dirt or foreign matter from the specimen, [ii].Clean the specimen with a solvent or cleaner and dry, steel specimen with residual magnetism should be demagnetized before use, [iii].Weigh the specimen to the nearest 0.001gm.[iv].Fix the specimen inside specimen holder,[v].Check and ensure the sand flow knob is in “OFF” position and loading lever is up,[vi]. Fill the sand inside in the hopper,[vii]. Switch “ON” the power switch, [viii].Press the motor “START” switch. Smoothly rotate Dimmerstat to adjust the RPM in clockwise direction and set the required motor RPM value and check the RPM by tachometer,[ix].Release the loading lever by turning the lever lifter down so that the specimen is in contact with the rubber wheel. Place the desired load on the loading pan,[x].The dead weight exerted on rubber wheel by loading lever is 30N. To conduct tests as per ASTM procedure two dead weight’s 4.07kg & 0.61kg are provided,[xi].“TURN ON” the sand flow knob to “ON” position. The sand flow rate through nozzle provided is in the range of 345
The rubber wheel will rotate up to the preset rpm value. {The motor will be switched OFF.},[xii]. Turn the sand flow knob to “OFF” position,[xiii]. Lift the loading lever to “UP” position and remove the weights from the loading pan. After test the loading lever near specimen will be hot, allow the sample to cool till it attains ambient temperature,[xv]. Measure the weight of the sample after the test,[xvi]. The loss in volume is computed in the following manner[202].

Loading lever ratio is 1: 2.5
Load exerted by loading lever with out dead weights is 30N
Wear out can be calculated by using the following formula
Specific Wear rate calculations:
1. Specific Wear rate in mm³/ N-m = [(Wt before test(g) - Wt after test (g)]x1000
   Density (gm/cm³) X Sliding distance
1. Percentage of wear out = (w1-w2)*100 =Δw*100------------------------------------------(2)
2. Sliding wear rate ( SWR) = (Δw*1000)/( ρ*S* )--------------------------------------------- (3)

Where ρ =density, S= sliding distance
W1-weight of specimen before test in gms,W2-weight of specimen after test in gms
ΔW=Mass lose due to abrasive wear in gms
Dry abrasive testing has been conducted for various un-coated and bond coated specimens. are shown in figure.5.2 and wear test results are discussed in chapter-8.

5.5. Characterization of thermal barrier coatings:

Once the TBC powder satisfies above characteristics discussed in 4.1.1 to 4.1.5, it is plasma spray coated on engine combustion chamber component metal substrates subjected to various mechanical tests to identify the good stability of coatings. All the relevant coating characteristics have been experimentally investigated and the obtained results are thus discussed and presented in this thesis. Following are the characterization of thermal barrier coating tests.

5.6. Plasma Spray coating:

Two types of substrates: flat plates 100 mm x 100 mm x 5 mm, and stainless steel (SS304), Aluminum alloy (Al-13Si) and cast iron were used as substrates. Typical
photographs of intermetallic bond coated and plasma spray Zirconated top coated substrates are shown Figure- 5.3(a)(b).

Figure 5.3(a). Bond and $\text{La}_2\text{Zr}_2\text{O}_7$ Top Coated engine component material substrates
Figure 5.3(b). Bond and $\text{Pr}_2\text{Zr}_2\text{O}_7$ Top Coated engine component material substrates

Two types of substrates: flat plates 100 mm x 100 mm x 5 mm, and stainless steel (SS304), Aluminum alloy (Al-13Si) and cast iron were used as substrates. Typical photographs of intermetallic bond coated and plasma spray Zirconated top coated substrates are shown Figure- 5.3(a)(b).

5.7. Thermal Shock Resistance Test: Thermal Shock Resistance is an ability of material to withstand sharp changes in temperature. If a ceramic material is rapidly cooled, its surface reaches the temperature of cooling environment and tends to contract (thermal contraction). Since the interior regions of the material are still hot, thermal contraction of the skin surface is impossible. This leads to formation of tensile stress (thermal stress) in the skin. Such thermal stresses may cause cracks and consequent failure. The TSRT has been conducted on metallic bond and ceramic top coated various engine component material substrates, to identify the failure of TBC coating before
coating on real combustion chamber components of a diesel engine. Thermal shock resistance of a material may be estimated in accordance to the formula:

\[ Rs = \frac{\lambda \sigma_f}{\alpha E} \]  

Where

Rs – Thermal shock resistance; \( \lambda \) - Thermal conductivity; \( \sigma_f \) – flexural strength, \( \alpha \) - coefficient of thermal expansion (CTE); E – Modulus of elasticity

A photograph of test rig can be viewed below figure 5.5, and its operation is explained by the following figure 5.5. Also figure 5.5. Shows the Complete experimental setup of TSRT with coated over SS specimen and the experimental details are explained in following.

The Thermal Shock Resistance (TSR) which has been conducted on standard TSR Test Rig the facility available in the college, The system has to be operated manually and the schematic representation of the test rig is shown 5.5, it consists of the following parts,
1. Rotary table for mounting the specimen, 2. Outer table frame for mounting torch and hose pipe. 3. Blower setup.

The rectangular metal table was designed for mounting the specimen such that the table can be rotated about the centre so that the TBC specimen can be brought into the oxyacetylene torch and hose pipe direction alternately for heating and cooling. The height of the table can be adjusted centrally by elevating screw, such that the nozzle of the torch is directed exactly at the centre of the specimen. In this position the torch is perpendicular to the TBC specimen avoiding direct exposure of the metal substrate of the coated specimen with the high temperature flame. The refractory bricks are placed on the rotary table, in order to avoid the influence of the Oxy-Acetylene flame on the table. The table is fully covered by placing high temperature resistance bricks, here 3 bricks are placed in the horizontal and vertical position and are then covered with a layer of ceramic wool of suitable thickness. This wool is chosen as it has very poor thermal conductivity.
The most widely used test for evaluation of thermal shock resistance (TSR) of TBCs are a) burner rig test b) simulated burner rig test and c) furnace test [203]. In the present study simulated burner rig test has been used to determine thermal shock resistance of coatings. The complete facilities simulated burner rig test has been used. The specimens were mounted parallel to the work spot at a height of ~ 50mm so that the heat radiated from the metal would be dissipated away from it. In this test, the central regions of the coated surfaces were targeted perpendicularly by oxy-acetylene flame. The orifice of the gun nozzle was maintained at a distance of 100-125mm from the coatings. And uniform gas pressures were maintained in order to achieve the desired high temperatures (1000\(^\circ\)C-1600\(^\circ\)C). The surface temperature was measured using Pt-Pt-13\%Rh thermocouple interface with digital indicator. The metal substrate was not exposed to high temperature. Since the flame was perpendicular to the ceramic plasma sprayed TBC surface, the amount heat flows was across the thickness of the ceramic coating. This ensured that if the bond coat got oxidized, it was only due to the flame source focused well within the ceramic surface and the metal surface was not exposed to the flame.

The surface of the coating experienced a temperature distribution from the middle of the substrate to the edges (1000\(^\circ\)C ± 20\(^\circ\)C in the middle, 500\(^\circ\)C at the edges). The accuracy of temperature measured by the thermocouple was ± 5\(^\circ\)C. Within about a minute the ceramic surface is heated up to 1200-1450\(^\circ\)C, while due to the backside cooling the substrate material is 200-300\(^\circ\)C colder by the cooled air. And again withdrawn the cooled substrate nearer to heating zone. After attaining the desired maximum temperatures, the assembly was held at these temperatures for durations of 1 minute. This is equivalent to subjecting the ceramic to thermal shock using the flame and soaking by cooled air supplied externally by the blower for duration of 1 minute. After several cycles, propagating cracks may have reached a stage where the topcoat intak/spalls off [203,204] Cycles of this kind are repeated until spallation of parts of the ceramic layer occurs, subsequently the samples were suddenly withdrawn from the flame and quenched in the ambient (without any additional source of cooling) [205-207].
After these tests the samples are inspected and analyze its phase analysis by XRD, microstructure by SEM, and chemical composition by EDAX. These studies give insight into the reasons for sample failure. Oxidation of the metal substrate, sintering of the layer, phase transitions and crack growth play the important roles in this process. Also the results from a theoretical analysis of the stress states within the layers and of the crack growth help to understand the experimental observations.

The most critical combinations in the experiment have been found to be 1000 cycles at 1450°C in the case of Alloy steel (SS-304) and Cast iron. The selected combinations were 850 cycles at 1350°C in the case of Aluminum alloy.

On completion of the characterization of the powders and the coatings by the above methods, the compositions which held out a promise of being competitive in its properties were subjected to further rigorous evaluation by coating them on actual engine components and the performance were thus evaluated.

5.7.1. Thermal barrier test:

The experiment for temperature drop across the coating determination was carried out in the simulated burner rig facility with slight modifications. In this set up, the test specimen was mounted perpendicular to the work spot. A torch of oxy-acetylene flame temperature say 1000°C ± 5°C was focused perpendicularly onto the centre of the coated sample and held for about twenty-thirty minutes. After the attaining steady state, the temperature drop across the coating was determined by measuring the temperature of the metal side using K-type thermocouple fixed to the centre of the sample with high temperature conducting Ag-Pd paste and also with non contact type infrared thermocouple (for particular thickness of 250-350µm coating). It is a measure of the ability of the coatings to act as TBCs on a comparative basis. The same procedure has been followed for determination of other temperature (900, 1100 and 1145°C) differences across the coating. The flux of heat through a material depends on the thermal gradient across the material and can be expressed [205].
5.7.2. Hardness Test:

The micro hardness test Experimental detailed procedure has been briefly discussed in the above section.5.2.2, on the ceramic topcoat was conducted by using MICROMET-3, Micro hardness tester (BUEHLER Ltd, USA). Three points were selected on each specimen to examine micro hardness for the as-sprayed and thermal shock tested specimens of SS-304 and Al-124 Aluminum Alloy, at 200 grams loading. The obtained results are discussed in the subsequent chapter 8.